COMPARISON OF ELEVATED TEMPERATURE TENSILE PROPERTIES AND FATIGUE BEHAVIOR OF TWO VARIANTS OF A WOVEN SiC/SiC COMPOSITE

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ABSTRACT

Tensile properties (elastic modulus, proportional limit strength, in-plane tensile strength, and strain at failure) of two variants of a woven SiC/SiC composite, manufactured during two separate time periods (9/99 and 1/01), were determined at 1038 and 1204°C by conducting tensile tests on specimens machined from plates. Continuous cycling fatigue tests ($R = 0.05$ and 20 cpm) were also conducted at the same two temperatures on specimens from both composites. In this study, average tensile properties, 95% confidence intervals associated with the tensile properties, and geometric mean fatigue lives of both composite materials are compared. The observed similarities and differences in the tensile properties are highlighted and an attempt is made to understand the relationship, if any, between the tensile properties and the fatigue behaviors of the two woven composites.

INTRODUCTION

Advanced gas turbine engines, which are typically designed to operate at augmented efficiencies, require higher operating temperatures. In order to design for such an environment, materials that can withstand these higher operating temperatures as well as provide sustained performance are required. Ceramic matrix composites (CMCs) are strongly preferred for these tasks because of their high temperature capability and high specific strength. A woven SiC/SiC composite (manufactured by a slurry-cast, melt-infiltration process) has been identified as a potential combustor liner material for the advanced aircraft gas turbine engines[1]. For design purposes, both the reliability and reproducibility of this woven CMC need to be established and suitable tensile property and fatigue life data bases are required.

The two woven composites evaluated in this study were manufactured by General Electric Power Systems Composites, LLC during September 1999 (9/99) and January 2001 (1/01) with the Chemical Vapor Infiltrated (CVI) SiC/slurry-cast/melt-infiltration process. Tensile properties and fatigue behavior (both without and with hold-time) at 1038 and 1204°C

for the 9/99 composite were previously reported[2]. Tensile properties of both the 9/99 and 1/01 composites at 816 and 1204°C and the variability exhibited by each of those properties were also documented earlier[3]. In this investigation, additional tensile tests at 1038°C and fatigue tests at 1038 and 1204°C were conducted on the 1/01 composite material. The tensile properties and fatigue lives from the 9/99 and 1/01 woven SiC/SiC composites generated at 1038 and 1204°C were compared to identify similarities as well as differences. In particular, the dependency of fatigue lives on the tensile properties of the woven composites was explored.

EXPERIMENTAL DETAILS

Sylramic™ fiber in a 5HS weave, 20 EPI configuration with a $[0/90]_{4S}$ lay-up constituted the fiber pre-form for both the 9/99 and 1/01 composites. In the case of the 1/01 composite, before matrix densification, the fiber pre-forms were subjected to an in-situ BN (iBN) heat treatment. Chemical vapor infiltrated, silicon-doped BN interphase and a CVI SiC matrix further subjected to a slurry-cast, melt infiltration process (MI) were utilized for both the composites. Rectangular composite plates (229 mm length, 152 mm width, and 2 mm thickness) were produced by the manufacturing process and test specimens for both tensile and fatigue tests were machined from these plates. The test section within these specimens was 10.2 mm wide and 28 mm in length. Additional details on the test specimen geometry and test system, including test specimen gripping method, test frame alignment procedure, specimen heating and temperature measurement techniques, strain measurement method, and test procedures, were reported previously[2,4,5]. In fatigue tests, triangular waveform with a frequency of 0.33 Hz (20 cpm) and an R-ratio (minimum load to maximum load) of R = 0.05 were used. In all the fatigue tests, failure was defined as separation of the test specimen into two pieces.

TENSILE PROPERTIES

Tensile properties investigated in this study are the following: elastic modulus, E; proportional limit strength (0.005% offset), PLS; in-plane tensile strength, ITS; and strain at failure, SF. Mean values and associated standard deviations of these four tensile properties are shown in Tables I and II, respectively, for the 9/99 and 1/01 composites. A total of 24 tensile tests were conducted at 1204°C for both the 9/99 and 1/01 composites. At 1038°C, due to a limited number of specimens, only six and eight tests were conducted for the 9/99 and 1/01 composites, respectively. Mean ITS and SF values of the 1/01 composite were significantly higher than the corresponding values for the 9/99 composite. No clear cut differences were observed among the mean E and PLS values between the two composites. In general, at both temperatures, standard deviations of the 1/01 material were higher than those exhibited by the 9/99 material with two exceptions (PLS at 1204°C and ITS at 1038°C).

Table I. Means and Standard Deviations of Tensile Properties for 9/99 MI SiC/SiC Composite

Temperature [°C]	Number of Tests [n]	E [GPa]	PLS [MPa]	ITS [MPa]	SF [%]
1038	6	209 {15}*	168 {20}	325 {29}	0.44 {0.06}
1204	24	182 {14}	166 {28}	307 {21}	0.46 {0.07}

* { } Denotes Standard Deviation

Table II. Means and Standard Deviations of Tensile Properties for 1/01 MI SiC/SiC Composite

Temperature [°C]	Number of Tests [n]	E [GPa]	PLS [MPa]	ITS [MPa]	SF [%]
1038	8	185 {27}*	162 {35}	426 {19}	0.60 {0.11}
1204	24	184 {25}	155 {28}	399 {37}	0.57 {0.12}

* { } Denotes Standard Deviation

Average tensile properties and corresponding 95% confidence intervals (error bars) at 1038 and 1204°C for the two composites are compared directly with bar charts in Figs. 1 to 4. The confidence intervals were estimated as twice the standard deviation for each tensile property. Student's t-test with a risk level, $\alpha = 0.02$, and a two-tailed distribution was used to compare mean values of all the tensile properties for both composites. At the investigated temperatures, no statistically significant differences were found to exist between the two composites for the mean values of E and PLS. Differences in the mean ITS and SF values were found to be statistically significant between the two composites. Higher scatter exhibited by the mean tensile properties of the 1/01 composite in comparison to the 9/99 composite and the statistically significant differences observed among the mean ITS and SF values of both composites might be due to the iBN heat treatment given to the fiber pre-forms of the 1/01 composite.

FATIGUE BEHAVIOR

Fatigue tests were conducted with a maximum stress, σ_{max}, of 179 MPa and an R-ratio, R, of 0.05 on specimens from both composites. The selected maximum stress was above the average PLS values of the two composites at both temperatures (Tables I and II). Three tests were conducted at each temperature to obtain a representative fatigue life. The observed geometric mean fatigue lives (equivalent to arithmetic mean fatigue lives in log space) are listed in Table III. For the 9/99 and 1/01 composites, the geometric mean fatigue life decreased as temperature increased from 1038 to 1204°C. At both temperatures, geometric mean fatigue lives of the 1/01 composite were higher than those of the 9/99 composite. Since average PLS values of both composites were very similar, the higher fatigue lives of 1/01 composite could have resulted from the higher average in-plane tensile strengths of this composite.

Table III. Geometric Mean Fatigue Lives of MI SiC/SiC Composite (σ_{max} = 179 MPa; R = 0.05)

Temperature [°C]	Number of Tests [n]	9/99 [Cycles]	1/01 [Cycles]
1038	3	17 173	32 028
1204	3	4 093	20 405

Specimens fatigued to failure at both 1038 and 1204°C are shown in Figs. 5 and 6. In general, a majority of the specimens failed in the test section with only two specimens failing in the transition region between the gripping area and the test section (Fig. 5). Fatigue life data from these two specimens were included in calculating the geometric mean fatigue life because of the following reasons: 1) failure locations were closer to the test section than the grip section

indicating that temperatures at these locations were very close to the test temperatures and 2) the large transition radius used in the specimen design, between the grip and test sections, did not significantly increase the dimensions at the failure locations. Arithmetic mean logarithmic cyclic lives and associated minimum and maximum values are plotted in Fig. 7 for both composites. For the limited number of fatigue tests conducted, fatigue lives of 1/01 composite exhibited much less scatter at both temperatures than those from the 9/99 composite. This observed trend in fatigue lives was opposite to that exhibited by the tensile properties for both the composites. Note that more tensile tests than fatigue tests were conducted on each composite and the scatter in the fatigue lives of the 1/01 composite might eventually increase if more fatigue tests were conducted.

DISCUSSION

The primary difference in processing between the two composites was the iBN heat treatment provided for the fiber pre-forms of the 1/01 composite before matrix densification. Similar treatment was not given to the fiber pre-forms of the 9/99 composite. As evidenced by the Student's t-test results of mean tensile properties of both composites, the iBN treatment seems to improve the mean ITS and SF values while having no significant influence on the mean E and PLS values. Moreover, the 1/01 composite, which received this treatment, exhibited much larger variation in all four tensile properties than the 9/99 composite. The higher geometric mean fatigue lives observed for the 1/01 composite as compared to the corresponding lives of the 9/99 composite correlated well with higher mean ITS values exhibited by the 1/01 composite. The reason for lower scatter observed in the fatigue lives of the 1/01 composite, which is opposite to the trend observed in the tensile properties, is not very clear due to the limited number of fatigue tests conducted in this study. A systematic fatigue investigation that includes at least six to ten specimens per test condition is required to quantify scatter in the fatigue data in a reliable manner.

CONCLUDING REMARKS

Tensile properties and fatigue behavior of two variants of a woven SiC/SiC composite manufactured during two separated time periods (9/99 and 1/01) were investigated at 1038 and 1204°C. Fiber pre-forms of the 1/01 composite were iBN heat-treated prior to matrix densification by slurry-cast, melt-infiltration process. No such treatment was given to the fiber pre-forms of the 9/99 composite. Average tensile properties and associated standard deviations were reported and compared for both composites. Geometric mean fatigue lives of both the composites are presented along with the associated scatter bands. The 1/01 composite exhibited higher average values of in-plane tensile strength and strain at failure in the tensile tests and higher geometric mean fatigue lives compared to the 9/99 composite. The 1/01 composite exhibited more variation in the tensile properties than the 9/99 composite, whereas the opposite was true for the scatter observed in the fatigue lives.

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REFERENCES

[1]D. Brewer, "HSR/EPM Combustor Materials Development Program," *Materials Science and Engineering,* **A261**, pp. 284-291, 1999.

[2]S. Kalluri, A. M. Calomino, and D. N. Brewer, "High Temperature Tensile Properties and Fatigue Behavior of a Melt-Infiltrated SiC/SiC Composite," *Fatigue 2002*, Proc. of the 8th Intl. Fatigue Congress, Vol. 3/5, A. F. Blom, Ed., Stockholm, Sweden, pp. 1965-1972, 2002.

[3]S. Kalluri, A. M. Calomino, and D. N. Brewer, "An Assessment of Variability in the Average Tensile Properties of a Melt-Infiltrated SiC/SiC Composite," *Ceramic Engineering and Science Proceedings*, Vol. 25, Issue 4, 28th International Conference on Advanced Ceramics and Composites: B, Edgar Lara-Curzio and Michael J. Readey, Eds., pp. 79-86, 2004.

[4]M. J. Verrilli, A. M. Calomino, and D. N. Brewer, "Creep-Rupture Behavior of a Nicalon/SiC Composite," *Thermal and Mechanical Test Methods and Behavior of Continuous-Fiber Ceramic Composites, ASTM STP 1309*, M. G. Jenkins, S. T. Gonczy, E. Lara-Curzio, N. E. Ashbaugh, and L. P. Zawada, Eds., American Society for Testing Materials, pp. 158-175, 1997.

[5]M. J. Verrilli, A. Calomino, and D. J. Thomas, "Stress/Life Behavior of a C/SiC Composite in a Low Partial Pressure of Oxygen Environment, Part I: Static Strength and Stress Rupture Database," 26th Annual Conf. on Composites, Advanced Ceramics, Materials, and Structures: A, *Ceramic Engineering and Science Proceedings*, Vol. 23, Issue 3, H.-T. Lin and M. Singh, Eds., pp. 435-442, 2002.

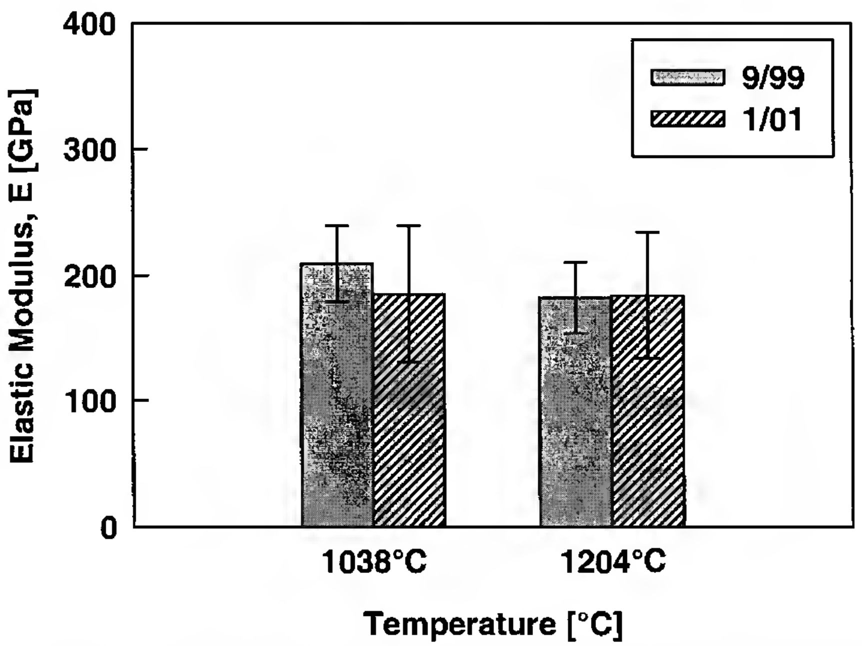

Figure 1: Average Elastic Moduli and 95% Confidence Intervals for Two Variants of a Woven SiC/SiC Composite

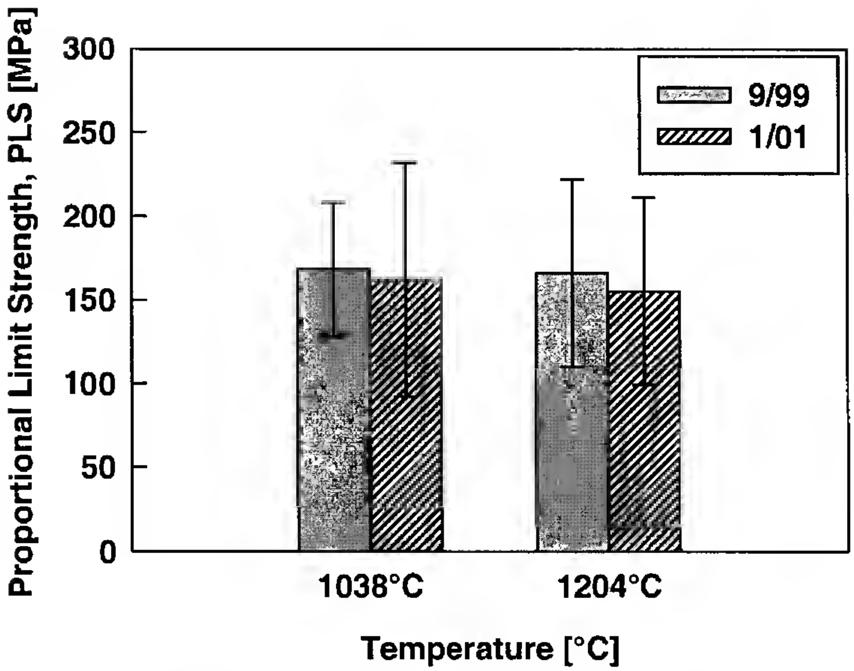

Figure 2: Average Proportional Limit Strengths and 95% Confidence Intervals for Two Variants of a Woven SiC/SiC Composite

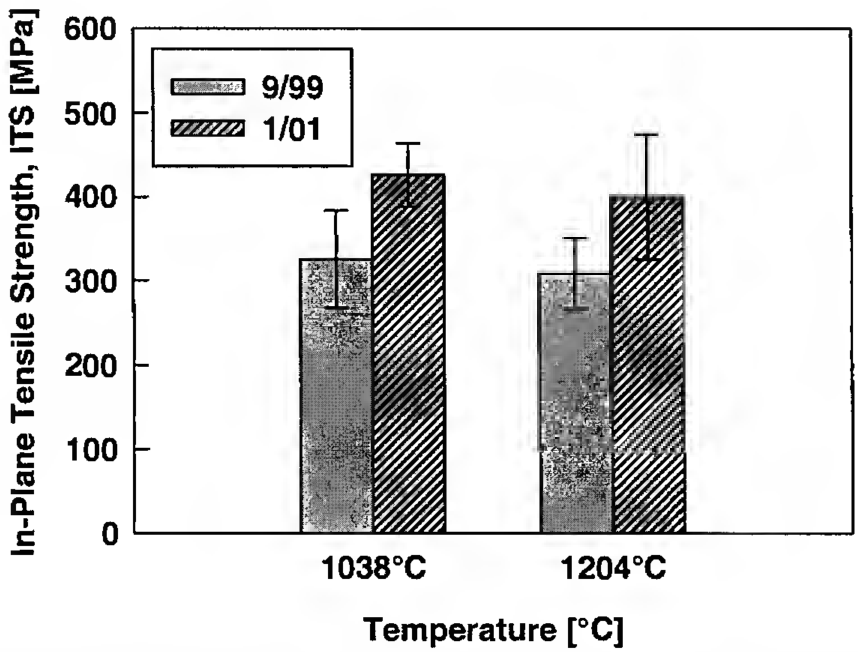

Figure 3: Average In-Plane Tensile Strengths and 95% Confidence Intervals for Two Variants of a Woven SiC/SiC Composite

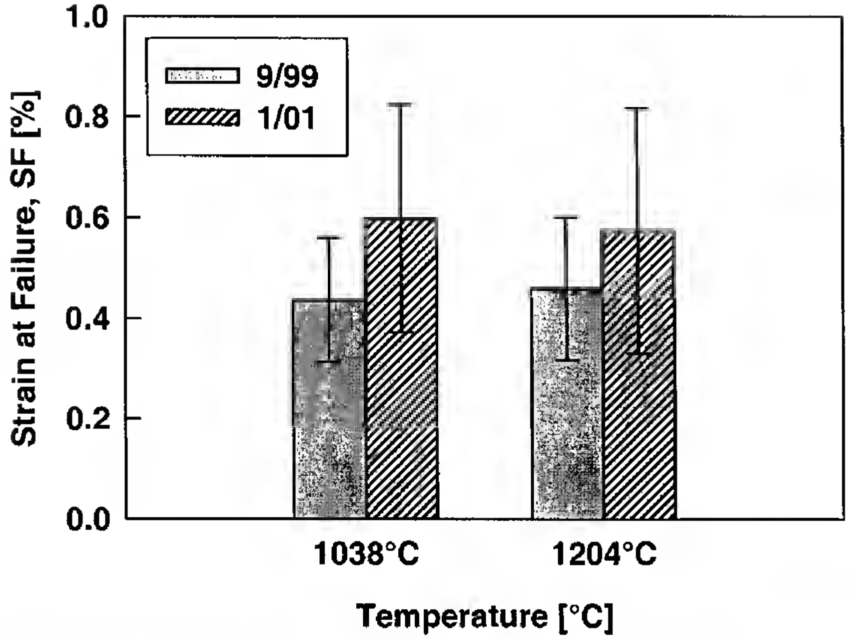

Figure 4: Average Strains at Failure and 95% Confidence Intervals for Two Variants of a Woven SiC/SiC Composite

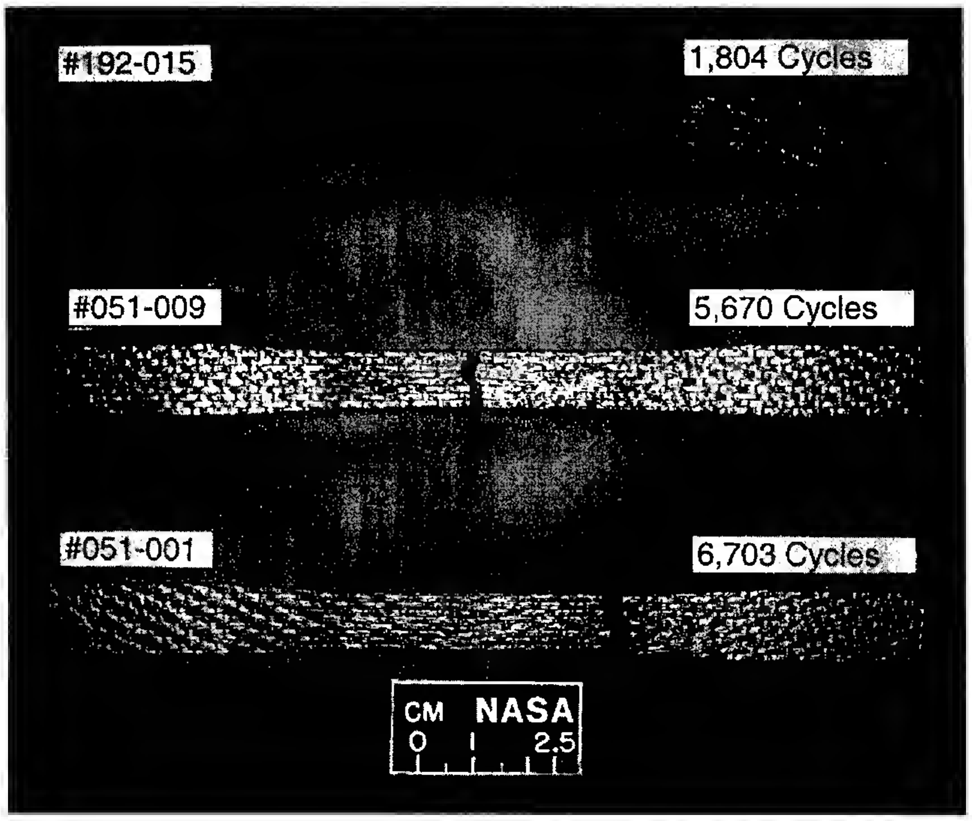

Figure 5: Specimens Tested in Fatigue at 1204°C: 9/99 SiC/SiC Composite

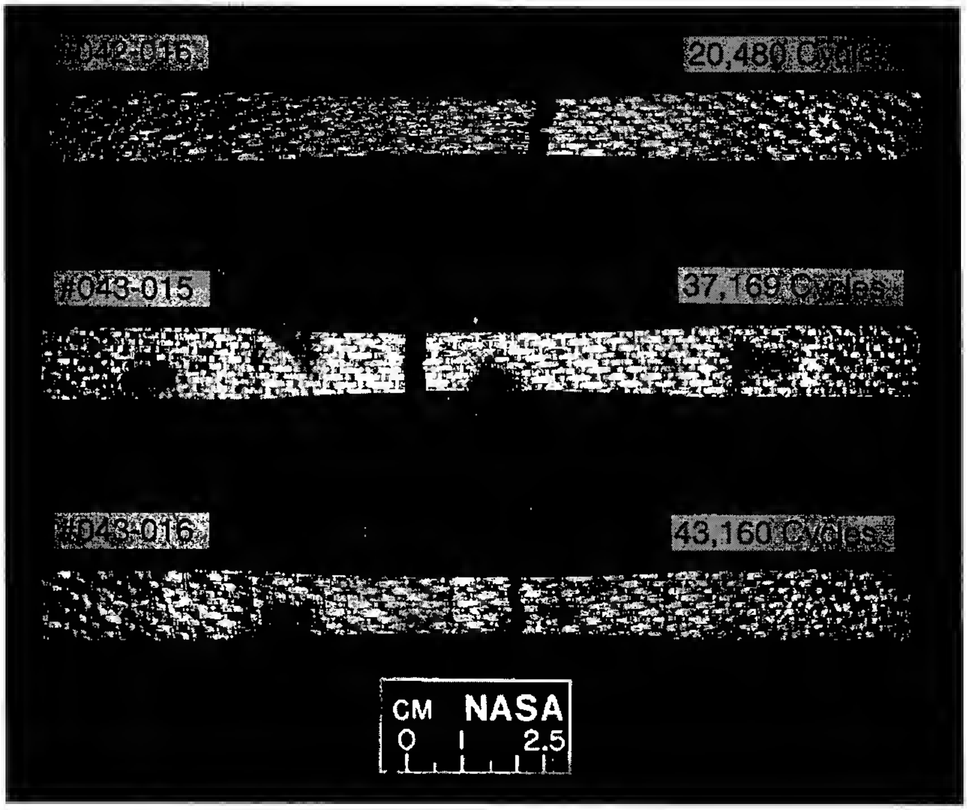

Figure 6: Specimens Tested in Fatigue at 1038°C:
1/01 SiC/SiC Composite

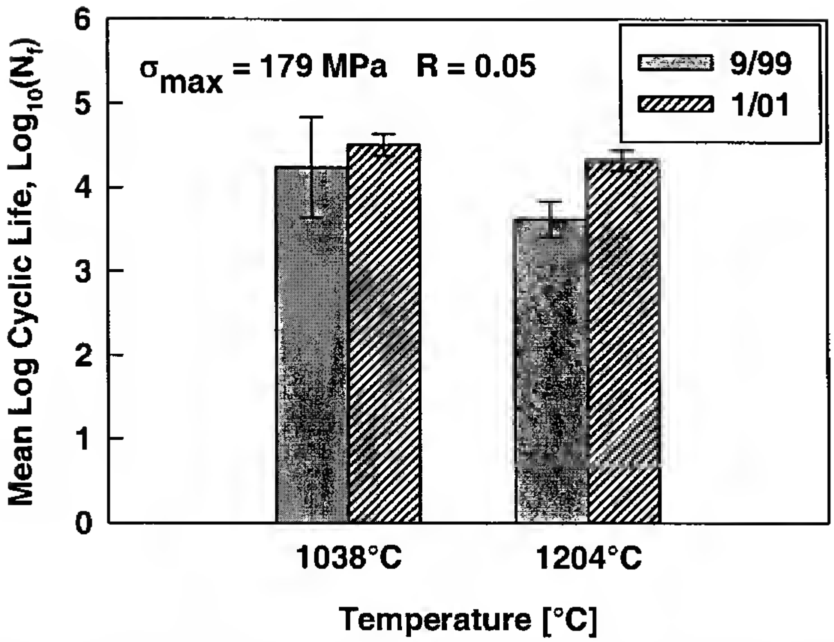

Figure 7: Arithmetic Mean Logarithmic Fatigue Lives and Extreme Values for Two Variants of a Woven SiC/SiC Composite